

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US04/041656

International filing date: 09 December 2004 (09.12.2004)

Document type: Certified copy of priority document

Document details: Country/Office: GB
Number: 0329192.9
Filing date: 17 December 2003 (17.12.2003)

Date of receipt at the International Bureau: 10 January 2005 (10.01.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

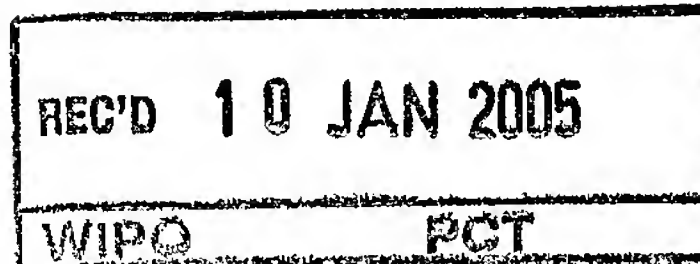
09 DEC 2004

PCTAUS 04 / 41656



INVESTOR IN PEOPLE

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ



I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

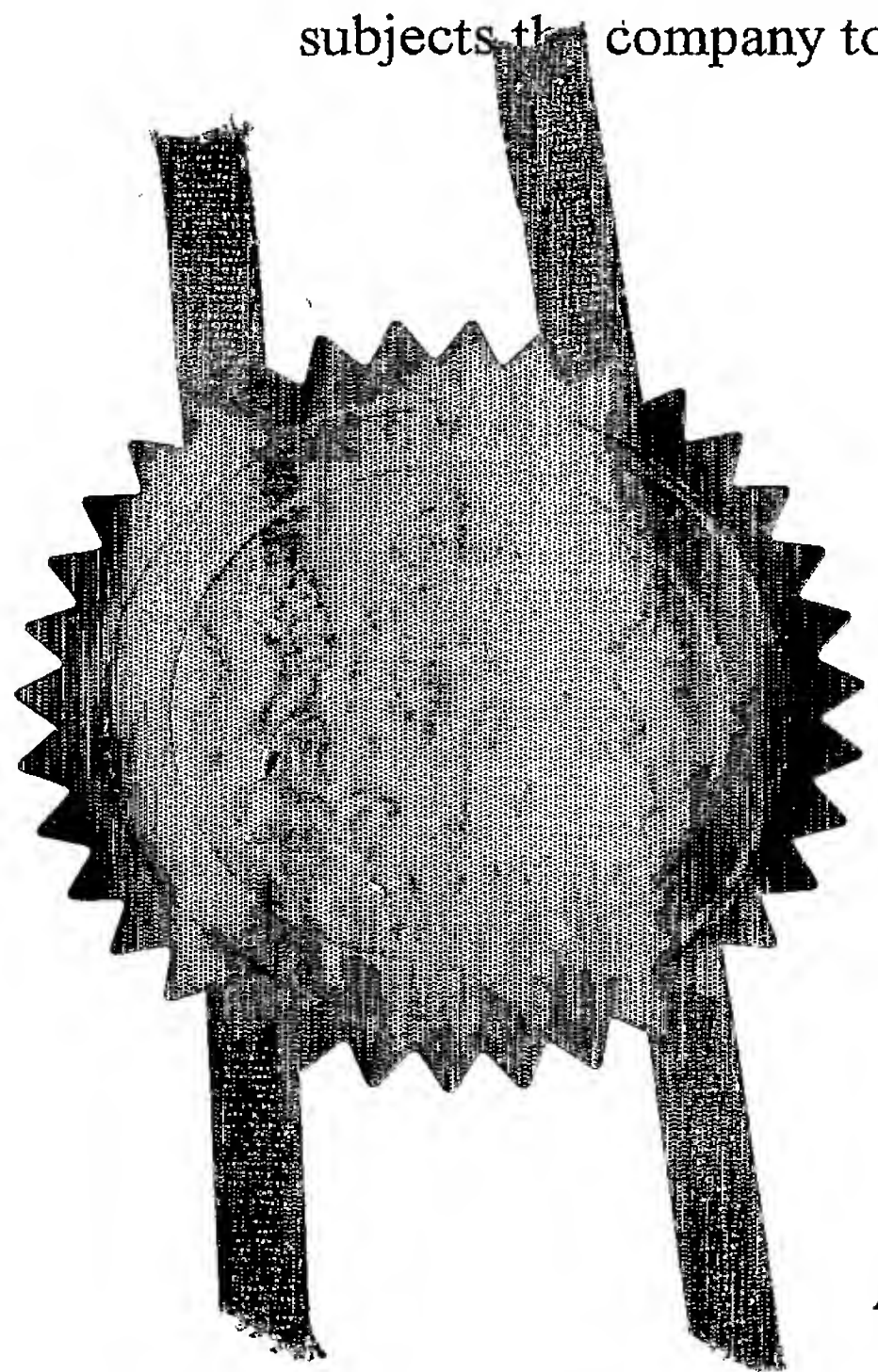
In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed

Andrew Gersey

Dated 29 September 2004



The
Patent
Office

PATENT OFFICE

17 DEC 2003

NEWPORT

17 DEC 2003-EP60047-1 D02914

01/7700/0.00-0329192.9 ACCOUNT CHA

The Patent Office

Cardiff Road
Newport
South Wales
NP9 1RH

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

1. Your reference

SN142

0329192.9

17 DEC 2003

2. Patent application number

(The Patent Office will fill in this part)

3. Full name, address and postcode of the or of each applicant (*underline all surnames*)

DOW CORNING CORPORATION
Midland
Michigan 48611
USA

Patents ADP number (*if you know it*)

8506040002

If the applicant is a corporate body, give the country/state of its incorporation

USA/Michigan

4. Title of the invention

FOAM CONTROL COMPOSITIONS

5. Name of your agent (*if you have one*)

P V Davies

"Address for service" in the United Kingdom to which all correspondence should be sent (*including the postcode*)

DOW CORNING LIMITED
Intellectual Property Department
Cardiff Road
Barry
CF63 2YL

Patents ADP number (*if you know it*)

414037001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (*if you know it*) the or each application number

Country

Priority application number
(*if you know it*)

Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (*Answer 'Yes' if:*

Yes

- a) any applicant named in part 3 is not an inventor, or
 - b) there is an inventor who is not named as an applicant, or
 - c) any named applicant is a corporate body.
- See note (d))

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description 19

Claim(s) 2

Abstract

Drawing(s) *RM*

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*) 1

Request for substantive examination (*Patents Form 10/77*)

Any other documents 1 fee sheet
(*please specify*)

11. I/We request the grant of a patent on the basis of this application.

Signature *P V Davies*
P V Davies

Date
16/12/2003

12. Name and daytime telephone number of person to contact in the United Kingdom P V Davies 01446 723505

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.*
- Write your answers in capital letters using black ink or you may type them.*
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.*
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.*
- Once you have filled in the form you must remember to sign and date it.*
- For details of the fee and ways to pay please contact the Patent Office.*

FOAM CONTROL COMPOSITIONS

[0001] This invention is concerned with foam control compositions for use in aqueous compositions which are liable to foam. The foam control compositions of the invention can
5 be added to detergent compositions, particularly detergent powders, to inhibit excessive foaming when the detergent is used in washing.

[0002] In many aqueous systems which are used e.g. in food processes, textile dyeing, paper production, sewage treatment and cleaning applications, the production of foam needs
10 to be controlled or prevented. It is important to keep the foam formation to an acceptable level when laundering is performed in automatic washing machines, especially front loading machines. Excessive foam would cause overflow of the washing liquor onto the floor as well as reduction in the efficiency of the laundering operation itself. There is a move in the detergent industry towards the use of detergent compositions which will perform to a higher
15 efficiency than hitherto. There is a need to control foam from e.g. increased surfactant levels in the detergent compositions, use of surfactants which have a higher foam profile than traditional surfactants and changing laundering conditions. It is desirable to keep the addition level of foam control compositions to a minimum. There has therefore arisen a need to develop more efficient foam control compositions for incorporation into detergent
20 compositions.

[0003] The most successful foam control agents, especially for detergents, are based on silicones. Examples are described in US-A-5767053, US-A-6521586 and US-A-5387364. US-A-6521587 describes a foam control agent, comprising an organopolysiloxane material
25 having at least one silicon-bonded aralkyl substituent, a water-insoluble organic fluid, an organosilicon resin and a hydrophobic filler. It is preferred that the water-insoluble organic fluid is miscible with the organopolysiloxane fluid at the operating temperature of the foam control agent.

30 [0004] There has been some demand for foam control agents containing a reduced amount of organopolysiloxane. US-A-5693256 describes a foam control agent comprising 100 parts by weight of a water-insoluble organic liquid, from 0.1 to 20 parts by weight of a

first hydrophobic filler and 0.1 to 20 parts by weight of a second hydrophobic filler, which may be a siloxane resin, said hydrophobic fillers being insoluble in the water- insoluble organic liquid. EP-B-687724 describes a foam control agent comprising 100 parts by weight of a water-insoluble organic liquid, from 0.1 to 20 parts by weight of a hydrophobic filler that is insoluble in the organic liquid and 0.1 to 20 parts by weight of an organosilicon resin that is at least partially soluble in the organic liquid.

[0005] WO97/34983 describes a granular antifoam mixture that contains a foam controlling active substance consisting of 70-95% paraffin wax mixture and 5-30% ethylenebisstearamide (EBSA). The paraffin wax mixture is solid at room temperature and at least 35% by weight and less than 100% by weight becomes liquid at 40C. This composition is claimed to give improved foam regulating effectiveness in cold and medium water temperature washing conditions.

15 [0006] A foam control composition according to the invention comprises a non-silicon-containing organic liquid, a branched siloxane resin, a particulate filler which is insoluble in the organic liquid, and an additive composition of melting point 35 to 100°C comprising a substantially non-polar organic material which is miscible with the organic liquid.

20 [0007] The non-silicon-containing organic liquid can for example be a liquid hydrocarbon oil or a liquid vegetable oil. It may be a single liquid or it may be a mixture of one or more such liquids. At least part of the organic liquid should be liquid at 40°C and the organic liquid is preferably liquid at 25°C.

25 [0008] Preferred liquid hydrocarbon oils are mineral oils, especially hydrogenated mineral oil or white oil, or isoparaffinic oils. The hydrocarbon oil preferably has an aromatic content of less than 10% by weight. Examples of liquid hydrocarbon oils which are not fully liquid at 25°C but are liquid at higher temperatures are petroleum jelly or Vaseline®.

[0009] Examples of vegetable oils which can be used as the non-silicon-containing organic liquid include peanut oil, coconut oil, olive oil, cottonseed oil, rapeseed oil and linseed oil.

5 [0010] The branched siloxane resin and preferably consists of siloxane units of the formula $R'_aSiO_{4-a/2}$ wherein R' denotes a hydroxyl, hydrocarbon or hydrocarbonoxy group, and wherein a has an average value of from 0.5 to 2.4. It preferably consists of monovalent trihydrocarbonsiloxy (M) groups of the formula $R''_3SiO_{1/2}$ and tetrafunctional (Q) groups $SiO_{4/2}$ wherein R'' denotes a monovalent hydrocarbon group. The number ratio of M groups
 10 to Q groups is preferably in the range 0.4:1 to 2.5:1 (equivalent to a value of a in the formula $R'_aSiO_{4-a/2}$ of 0.86 to 2.15), more preferably 0.4:1 to 1.1:1 and most preferably 0.5:1 to 0.8:1 (equivalent to $a=1.0$ to $a=1.33$). The branched siloxane resin is preferably a solid at room temperature. The molecular weight of the resin can be increased by condensation, for example by heating in the presence of a base. The base can for example be an aqueous or
 15 alcoholic solution of potassium hydroxide or sodium hydroxide, e.g. a solution in methanol or propanol. A resin comprising M groups, trivalent $R''SiO_{3/2}$ (T) units and Q units can alternatively be used, or up to 20% of units in the branched siloxane resin can be divalent units $R''_2SiO_{2/2}$. The group R'' is preferably an alkyl group having 1 to 6 carbon atoms, for example methyl or ethyl, or can be phenyl. It is particularly preferred that at least 80%, most
 20 preferably substantially all, R'' groups present are methyl groups. The resin may be a trimethyl-capped resin. Other hydrocarbon groups may also be present, e.g. alkenyl groups present for example as dimethylvinylsilyl units, most preferably not exceeding 5% of all R'' groups. Silicon bonded hydroxyl groups and/or alkoxy, e.g. methoxy, groups may also be present.

25 [0011] The branched siloxane resin is preferably present at least 1%, more preferably at least 2% by weight based on the organic liquid, up to 40%, preferably up to 20%. For most antifoam uses it is preferred that the branched siloxane resin is present at 2 to 10% by weight based on the organic liquid.

30 [0012] The particulate filler is generally solid at 100°C and can for example be silica, preferably with a surface area as measured by BET measurement of at least 50 m²/g., titania,

ground quartz, alumina, an aluminosilicate, an organic wax, e.g. polyethylene wax or microcrystalline wax, zinc oxide, magnesium oxide, a salt of an aliphatic carboxylic acids, a reaction product of an isocyanate with an amine, e.g. cyclohexylamine, or an high melting (above 100°C) alkyl amide such as ethylenebisstearamide or methylenebisstearamide.

5 Mixtures of two or more of these can be used. The filler is preferably hydrophobic. Some of the fillers mentioned above are not hydrophobic in nature, but can be used if made hydrophobic. This could be done either in situ (i.e. when dispersed in the organic liquid), or by pre-treatment of the filler prior to mixing with the organic liquid. A preferred filler is silica which is made hydrophobic. Preferred silica materials are those which are prepared by
10 heating, e.g. fumed silica, or precipitation. The silica filler may for example have an average particle size of 0.5 to 50µm, preferably 2 to 30 and most preferably 5 to 25µm. It can be made hydrophobic by treatment with a fatty acid, but is preferably done by the use of methyl substituted organosilicon materials such as dimethylsiloxane polymers which are end-blocked with silanol or silicon-bonded alkoxy groups, hexamethyldisilazane, hexamethyldisiloxane or
15 organosilicon resins containing $(\text{CH}_3)_3\text{SiO}_{1/2}$ groups. Hydrophobing is generally carried out at a temperature of at least 100°C. Mixtures of fillers can be used, for example a highly hydrophobic silica filler such as that sold under the Trade Mark 'Sipernat D10' can be used together with a partially hydrophobic silica such as that sold under the Trade Mark 'Aerosil R972'.

20

[0013] The amount of hydrophobic filler in the foam control composition of the invention is preferably 0.5-50% by weight based on the organic liquid, more preferably from 1 up to 10 or 15% and most preferably 2 to 8%.

25 [0014] The additive composition of melting point 35 to 100°C is based on a substantially non-polar organic material which is miscible with the organic liquid. By 'miscible', we mean that materials in the liquid phase (i.e., molten if necessary) mixed in the proportions in which they are present in the foam control composition do not show phase separation. This can be judged by the clarity of the liquid mixture in the absence of any filler
30 or resin. If the liquids are miscible the mixture is clear and remains as one phase. If the liquids are immiscible the mixture is opaque and separates into two phases upon standing.

The non-polar organic material preferably has a melting point of at least 45°C. The non-polar organic material generally enhances the effectiveness of foam control achieved by the blend of organic liquid, branched siloxane resin and particulate filler.

5 [0015] One preferred type of substantially non-polar organic material is a polyol ester which is a polyol substantially fully esterified by carboxylate groups each having 7 to 36 carbon atoms. Other suitable non-polar organic materials include hydrocarbon waxes.

[0016] The polyol ester is preferably a glycerol triester or an ester of a higher polyol
10 such as pentaerythritol or sorbitol, but can be a diester of a glycol such as ethylene glycol or propylene glycol, preferably with a fatty acid having at least 14 carbon atoms, for example ethylene glycol distearate. Examples of preferred glycerol triesters are glycerol tripalmitate, which is particularly preferred, glycerol tristearate and glycerol triesters of saturated
15 carboxylic acids having 20 or 22 carbon atoms such as that sold under the Trade Mark 'Synchrowax HRC'. Alternative suitable polyol esters are esters of pentaerythritol such as pentaerythritol tetrabehenate and pentaerythritol tetrastearate. The polyol ester can advantageously contain fatty acids of different chain length, which is common in natural products. Most preferably the polyol ester is substantially fully esterified by carboxylate groups each having 14 to 22 carbon atoms. By "substantially fully esterified" we mean that
20 for a diol such as ethylene glycol or a triol such as glycerol, at least 90% and preferably at least 95% of the hydroxyl groups of the polyol are esterified. Higher polyols, particularly those such as pentaerythritol which show steric hindrance, may be "substantially fully esterified" when at least 70 or 75% of the hydroxyl groups of the polyol are esterified; for example pentaerythritol tristearate has the effect of a fully esterified polyol ester. The
25 additive composition can comprise a mixture of polyol esters, for example a mixture containing carboxylate groups of different carbon chain length such as glyceryl tristearate and glyceryl tripalmitate, or glyceryl tristearate and Synchrowax HRC, or ethylene glycol distearate and Synchrowax HRC. Foam control compositions containing mixtures of two polyol esters in the additive composition may give greater foam control efficiency than
30 compositions containing either polyol ester alone as the additive.

[0017] Examples of hydrocarbon waxes suitable as the non-polar organic material of melting point 35 to 100°C include paraffin wax, for example of melting point in the range 50 to 60°C, optionally blended with microcrystalline wax, for example the wax sold under the Trade Mark 'Cerozo'.

5

[0018] In general, each of the different types of non-polar organic material of melting point 35 to 100°C can be used with each type of organic liquid. For example a hydrocarbon wax can be used with a liquid hydrocarbon oil. The resulting composition contains the ingredients defined in WO97/34983 with the addition of a branched siloxane resin. We have found that the branched silicone resin significantly enhances the foam control achieved by the composition. Alternatively a polyol ester can be used with a liquid hydrocarbon oil, branched siloxane resin and hydrophobic filler, and this composition also shows enhanced foam control compared to the compositions of WO97/34983. Similarly either a polyol ester or a hydrocarbon wax can be used with a liquid vegetable oil.

15

[0019] The blend of organic liquid, branched siloxane resin, particulate filler and non-polar organic material of melting point 35 to 100°C may further contain a component which contains groups more polar than the groups present in the polyol ester non-polar organic material. The more polar group preferably contains an active hydrogen atom, that is one liable to undergo hydrogen bonding. Examples of more polar groups are unesterified -OH groups (alcohol or phenol groups), unesterified -COOH groups, amide groups or amino groups. The more polar component may have a melting point of at least 35°C, for example in the range 45-110°C, or may have a lower melting point, for example it may be liquid provided that the mixture of the non-polar and more polar components has a melting point of at least 35°C. The more polar component is preferably miscible with the polyol ester and may also be miscible with the liquid hydrocarbon polymer. Examples of more polar components are fatty alcohols, ethoxylated fatty alcohols, ethoxylated fatty acids, ethoxylated alkyl phenols and partial esters of polyols such as monoesters or diesters of glycerol and a carboxylic acid having 8 to 30 carbon atoms, for example glycerol monostearate, sorbitan monostearate, glycerol monolaurate or glycerol distearate, and mixtures thereof, alkyl phenols having one or more alkyl substituent and preferably containing a total of 6 to 12 carbon atoms

25

30

in the alkyl substituent or substituents attached to the phenol nucleus, for example octylphenol or nonylphenol or di(t-butyl)phenol, fatty acids having 8 to 36 carbon atoms, for example stearic acid, palmitic acid, behenic acid, oleic acid and/or 12-hydroxystearic acid, monoamides of fatty acids having 12 to 36 carbon atoms, for example stearamide, or alkyl
5 amines having 8 to 30 carbon atoms.

[0020] The substantially non-polar material of melting point 35 to 100°C and the more polar component can be present in weight ratio 5:95 to 95:5.

10 [0021] The additive composition, comprising the substantially non-polar material of melting point 35 to 100°C and optionally the more polar component, is preferably present in the foam control composition at 10-200% by weight based on the organic liquid, most preferably 20 up to 100 or 120%.

15 [0022] The foam control compositions according to the invention may be made by combining the organic liquid, the hydrophobic filler, the branched siloxane resin and the substantially non-polar material of melting point 35 to 100°C in any convenient way. The organic liquid, the hydrophobic filler and the organosilicon resin are preferably mixed together under shear. The non-polar additive can be present during this mixing or can be
20 added later, or part of the non-polar additive can be present during shear mixing and part can be added later. For example when the organic liquid is a hydrocarbon oil such as a paraffin oil, we have found that the best foam control results are achieved when the oil is blended with a paraffin wax, a branched siloxane resin and a filler and the resulting compound is mixed with a polyol ester of melting point between 40 and 90°C. Where the filler needs to be made
25 hydrophobic in situ, the manufacturing process includes a heating stage, preferably under reduced pressure, in which the filler and the treating agent are mixed together in part or all of the organic liquid in the presence of a suitable catalyst if required.

[0023] The foam control composition of the present invention may be supported on a
30 particulate carrier, particularly when the composition is to be used in a powdered product such as a detergent powder. Examples of carriers and/or supports are zeolites, for example

Zeolite A or Zeolite X, other aluminosilicates or silicates, for example magnesium silicate, phosphates, for example powdered or granular sodium tripolyphosphate, sodium sulphate, sodium carbonate, for example anhydrous sodium carbonate or sodium carbonate monohydrate, sodium perborate, a cellulose derivative such as sodium

5 carboxymethylcellulose, granulated starch, clay, sodium citrate, sodium acetate, sodium bicarbonate, sodium sesquicarbonate and native starch. The organic liquid containing the hydrophobic filler, the branched siloxane resin and the non-polar additive is preferably deposited on the carrier particles in non-aqueous liquid form, for example a temperature in the range 40-100°C. In an alternative process, the organic liquid, the hydrophobic filler, the
10 organosilicon resin and the additive composition are emulsified in water and the resulting aqueous emulsion is deposited on the carrier particles. The supported foam control composition is preferably made by an agglomeration process in which the foam control composition is sprayed onto the carrier particles while agitating the particles. The particles are preferably agitated in a high shear mixer through which the particles pass continuously. In
15 one preferred process, the particles are agitated in a vertical, continuous high shear mixer in which the foam control composition is sprayed onto the particles. One example of such a mixer is a Flexomix mixer supplied by Hosokawa Schugi.

[0024] The supported foam control composition may additionally include a water-
20 soluble or water-dispersible binder to improve the stability of the particles. Examples of binders are polycarboxylates, for example polyacrylic acid or a partial sodium salt thereof or a copolymer of acrylic acid, for example a copolymer with maleic anhydride, polyoxyalkylene polymers such as polyethylene glycol, which can be applied molten or as an aqueous solution and spray dried, reaction products of tallow alcohol and ethylene oxide, or cellulose ethers,
25 particularly water-soluble or water-swellaable cellulose ethers such as sodium carboxymethylcellulose, or sugar syrup binders. The water-soluble or water-dispersible binder can be mixed with the foam control composition before being deposited on the carrier, but preferably is separately deposited on the carrier particles. In one preferred procedure the foam control composition is deposited on the carrier particles as a non-aqueous liquid at a
30 temperature in the range 40-100°C and the water-soluble or water-dispersible binder is at the same time or subsequently, or at both times, deposited on the carrier from a separate feed as an aqueous solution or dispersion.

[0025] The supported foam control composition may optionally contain a surfactant to aid dispersion of the foam control composition in the binder and/or to help in controlling the "foam profile", that is in ensuring that some foam is visible throughout the wash without overfoaming. Examples of surfactants include silicone glycols, or fatty alcohol ether sulphate or linear alkylbenzene sulphonate, which may be preferred with a polyacrylic acid binder. The surfactant can be added to the foam control composition undiluted before the silicone is deposited on the carrier, or the surfactant can be added to the binder and deposited as an aqueous emulsion on the carrier.

10

[0026] The foam control composition can alternatively be provided in the form of an oil-in-water emulsion using any of the surfactants described in US-A-6521587. Alternatively the foam control agent can be provided as a water-dispersible composition in a water-dispersible vehicle such as a silicone glycol or in another water-miscible liquid such as ethylene glycol, polyethylene glycol, propylene glycol, a copolymer of ethylene glycol and propylene glycol, an alcohol alkoxylate, an alkoxyalkanol or hydroxyalkyl ether or an alkylphenol alkoxylate.

15

[0027] The foam control agents according to this invention are useful for reducing or preventing foam formation in aqueous systems, including foam generated by detergent compositions during laundering and foam generated in such processes as paper making and pulping processes, textile dyeing processes, cutting oil, coatings and other aqueous systems where surfactants may produce foam.

20

25 [0028] The following examples illustrate the invention. All parts and percentages are expressed by weight unless otherwise stated.

Example 1

30 [0029] 90% white mineral oil sold under the Trade Mark 'Kaydol' was blended with 6% 'Sipernat D10' (Trade Mark) hydrophobic treated silica and 4% of a MQ siloxane resin having a ratio of M groups to Q groups of 0.65 to form an antifoam composition C1.

[0030] 65% composition C1 was mixed with 35% glyceryl tristearate to form Antifoam Composition 1 of the present invention.

5 Example 2

[0031] 65% composition C1 was mixed with 35% Synchronowax HRC to form Antifoam Composition 2.

10 Example 3

[0032] 65% composition C1 was mixed with 35% paraffin wax of melting point 54-56°C to form Antifoam Composition 3.

15 Example 4

[0033] 25% Kaydol white mineral oil was blended with 75% paraffin wax of melting point 54-56°C, 4% of the MQ siloxane resin of Example 1 and 6% 'Sipernat D10'. 65% of the resulting composition was mixed with 35% Synchronowax HRC to form Antifoam
20 Composition 4.

Example 5

[0034] The procedure of Example 4 was repeated to form Antifoam Composition 5,
25 except that 40% Kaydol white mineral oil was initially blended with 60% of the paraffin wax.

[0035] In a comparative example, Example 4 was repeated to form antifoam composition C4, except that the Kaydol white mineral oil was wholly replaced by the paraffin wax of melting point 54-56°C.

Example 6

5 [0036] 90% rapeseed oil was blended with 6% Sipernat D10 and 4% of the MQ siloxane resin of Example 1 to form an antifoam composition C2.

[0037] 65% composition C2 was mixed with 35% glyceryl tristearate to form Antifoam Composition 6 of the present invention.

10 Example 7

[0038] 65% composition C2 was mixed with 35% Synchronax HRC to form Antifoam Composition 7.

15 Example 8

[0039] 65% composition C2 was mixed with 35% paraffin wax of melting point 54-56°C to form Antifoam Composition 8.

20 [0040] Antifoam Compositions 1 to 8 and comparative antifoam compositions C1, C4 and C2 were each sprayed onto sodium carbonate powder in a granulating mixer to produce a supported foam control composition containing about 16% of the active foam control composition.

25 Comparative Tests

[0041] Supported Antifoam Compositions 1 to 8 and supported comparative foam control agents C1 to C4 were tested in a powder detergent formulation which comprised 327 parts by weight zeolite, 95 parts of a 55% aqueous solution of sodium
30 dodecylbenzenesulphonate, 39 parts ethoxylated lauryl stearyl alcohol, 39 parts sodium sulphate, 125 parts sodium carbonate and 125 parts sodium perborate. Comparative foam control agent C3 was a commercially used foam control composition based on a polydiorganosiloxane fluid containing hydrophobic silica applied to a sodium carbonate

carrier. Each supported foam control composition was used at about 1% by weight of the detergent powder (0.15% by weight active foam control compound based on detergent powder). The evaluation was made in a Miele 934 front loading washing machine, loaded with 16 cotton towels, 100g of the detergent formulation, 17 litres of water of 9 degree

- 5 German hardness using a wash cycle of 65 minutes at 40°C or 95°C. The foam height was measured every 5 minutes during the wash cycle and recorded, where the value indicated is the foam height in the washing machine, with 100% referring to the fact that the window of the machine was full of foam, 50%, that is was half full of foam. The results are described in Table 1 below.

Table 1

			% Foam height with time (mins)													
Composition	% Active	Temp C	0	5	10	15	20	25	30	35	40	45	50	55	60	65
1	16.44	40	0	0	0	0	0	0	0	10	30	40	50	60	90	100
2	16.35	40	0	0	10	10	10	20	30	40	50	70	70	100	100	100
3	16.35	40	0	0	0	0	0	0	0	0	0	0	0	5	5	5
4	16.35	40	0	80	80	70	30	20	30	30	40	40	50	60	70	80
5	16.31	40	0	70	60	40	20	20	10	20	30	30	40	60	60	70
C4	15.87	40	0	100	100	100	100	100	100	100	100	100	100	100	100	100
6	16.24	40	0	0	20	30	40	60	90	100	100	100	100	100	100	100
7	16.33	40	0	10	20	30	60	70	100	100	100	100	100	100	100	100
8	16.35	40	0	0	0	0	10	20	30	60	70	90	100	100	100	100
1	16.44	95	0	0	0	0	0	0	0	0	10	20	30	40	50	50
2	16.35	95	0	0	0	0	10	10	0	0	0	20	30	50	50	50
3	16.35	95	0	0	0	0	0	0	0	0	0	10	20	35	45	45
4	16.35	95	0	80	80	40	20	0	0	0	0	0	10	20	20	30
5	16.31	95	0	60	20	0	0	0	0	0	0	0	10	20	20	30
C4	15.87	95	0	100	100	100	100	70	0	0	0	0	20	30	30	50

			% Foam height with time (mins)													
Composition	% Active	Temp C	0	5	10	15	20	25	30	35	40	45	50	55	60	65
6	16.24	95	0	0	20	50	60	50	30	10	30	40	60	70	70	100
7	16.33	95	0	10	20	40	50	50	30	10	20	40	60	60	70	100
8	16.35	95	0	0	0	0	0	0	0	0	30	50	90	100	100	100
C1	17.80	40	0	0	20	20	20	40	50	60	70	90	100	100	100	100
C2	18.17	40	0	20	50	70	90	90	100	100	100	100	100	100	100	100
C1	17.80	95	0	0	10	30	20	20	30	30	60	90	100	100	100	100
C2	18.17	95	0	10	50	80	90	100	100	100	100	100	100	100	100	100
C3	18.53	40	0	0	10	40	50	80	100	100	100	100	100	100	100	100
C3	18.53	95	0	0	10	30	50	60	100	100	100	100	100	100	100	100

[0042] The results for Antifoam Compositions 1 and 2 compared to C1 show that addition of a polyol ester such as a triglyceride to a mineral oil antifoam compound containing branched siloxane resin and hydrophobic silica filler just prior to granulation on a carrier enhances the foam control by the mineral oil antifoam compound in the 40°C wash cycle and especially the 95°C wash cycle.

[0043] The results for Antifoam Compositions 6 and 7 compared to C2 show that addition of a polyol ester such as a triglyceride to a vegetable oil antifoam compound containing branched siloxane resin and hydrophobic silica filler just prior to granulation on a carrier enhances the foam control by the vegetable oil antifoam compound in the 40°C wash cycle and especially the 95°C wash cycle.

[0044] The results for Antifoam Compositions 3 and 8 compared to C1 and C2 show that addition of a paraffin wax (melting point 54-56°C) to either the mineral oil antifoam compound or the rapeseed oil antifoam compound just prior to granulation on a carrier enhances the performance of each antifoam compound in both the 40°C wash cycle and the 95°C wash cycle. This is despite the fact that the wax is below its melting point throughout the whole of the 40°C wash and the first 30 minutes of the 95°C wash cycle.

[0045] The poor results shown by comparative foam control composition C4 confirm that the organic liquid must be present as well as wax for the compound to be an effective foam control agent. Using C4, foam control performance is initiated at or close to the onset of liquefaction of the wax at 30 mins into the 95°C wash cycle and no foam control performance in the 40°C wash cycle.

[0046] The Antifoam Compositions 4 and 5, where the compound contains a mixture of paraffin oil and paraffin wax in conjunction with MQ resin and hydrophobic silica (#4 and #5) with post-addition of a triglyceride prior to granulation onto a carrier, are especially preferred because the foam profile in the 40°C is relatively uniform through the wash cycle

and good foam control is also achieved at 95°C. Further modification of the foam profile can be achieved by varying the mineral oil and paraffin wax ratio in the compound.

Example 9

5

[0047] 90% of a paraffin wax liquid paraffin mixture was blended with 6% Sipernat D10 and 4% of the MQ siloxane resin of Example 1 to form Antifoam Composition 9. Antifoam Composition 9 was deposited onto a sugar carrier to produce a supported foam control composition.

10

Example 10

[0048] 65% Antifoam Composition 9 was mixed with 35% Synchronowax HRC to form Antifoam Composition 10.

15

Example 11

[0049] 65% Antifoam Composition 9 was mixed with 28% Synchronowax HRC and 7% octylphenol to form Antifoam Composition 11.

20

Example 12

[0050] 65% Antifoam Composition 9 was mixed with 35% glyceryl monostearate (90% pure) to form Antifoam Composition 12.

25

[0051] Antifoam Compositions 10 to 12 were each sprayed onto sodium carbonate powder in a granulating mixer to produce a supported foam control composition containing about 16% of the active foam control composition.

30

[0052] Supported Antifoam Compositions 9 to 12 and supported comparative foam control agents D1 to D3 were tested in comparative wash tests under the conditions described above. The comparative foam control agents were commercially used supported foam control compositions based on polydiorganosiloxane fluids containing hydrophobic silica. D1 and

D2 used a zeolite carrier and D3 used a starch carrier. The results are shown in Table 2 below.

Table 2

			% Foam height with time (mins)													
Composition	% Active	Temp C	0	5	10	15	20	25	30	35	40	45	50	55	60	65
9		40	0	100	100	100	30	10	10	20	30	30	40	50	60	60
10	16.56	40	0	70	55	45	15	10	10	15	20	25	30	35	45	45
11	16.40	40	0	40	20	10	0	0	0	0	10	10	20	20	30	30
10	16.56	95	0	90	90	90	80	50	10	0	0	0	10	10	30	30
11	16.40	95	0	70	40	20	10	0	0	0	0	10	20	30	40	40
12	16.37	40	0	70	90	80	50	70	70	80	100	100	100	100	100	100
D1	11.50	40	0	5	30	50	70	90	100	100	100	100	100	100	100	100
D2	11.12	40	0	0	0	0	0	0	0	5	20	25	35	45	50	50
D3	14.00	40	0	0	0	0	0	0	0	0	0	5	10	20	30	40
12	16.37	95	0	80	90	80	60	50	40	50	30	50	70	80	90	100
D1	11.50	95	0	0	40	60	90	90	100	100	100	100	100	100	100	100
D2	11.12	95	0	0	0	0	0	0	10	10	10	10	20	10	0	0
D3	14.00	95	0	0	0	0	0	0	0	50	100	100	100	100	100	100

[0053] The result for Composition 9 shows that an antifoam composition comprising a paraffin wax liquid paraffin mixture blended with hydrophobic silica and a branched siloxane resin of Example 1 is an effective antifoam, particularly in the later stages of the wash. The results for Compositions 10 and 11 show that addition of a non-polar polyol ester such as a glycerol triester further improves the foam control. Composition 10 is of particular interest as the foam profile obtained is very different from that obtained with current commercial foam control granules. A detergent manufacturer might consider that this better matches consumer foam profile preferences. The results for Composition 12 show that a more polar organic compound such as a glycerol monoester gives little improvement to an antifoam composition comprising a paraffin wax liquid paraffin mixture, hydrophobic silica and a branched siloxane resin although the more polar organic compound may improve the foam control of an antifoam composition comprising a liquid paraffin, a non-polar polyol ester, hydrophobic silica and a branched siloxane resin.

CLAIMS

1. A foam control composition comprising a non-silicon-containing organic liquid, a branched siloxane resin, a particulate filler which is insoluble in the organic liquid, and an additive composition of melting point 35 to 100°C comprising a substantially non-polar organic material which is miscible with the organic liquid.
2. A foam control composition according to Claim 1, characterised in that the organic liquid is a hydrocarbon oil.
3. A foam control composition according to Claim 1, characterised in that the organic liquid is a vegetable oil.
4. A foam control composition according to any of Claims 1 to 3, characterised in that the additive composition comprises a polyol ester which is a polyol substantially fully esterified by carboxylate groups each having 7 to 36 carbon atoms.
5. A foam control composition according to Claim 4, characterised in that the additive composition comprises 5-95 parts by weight of the organic polyol ester and 5-95 parts by weight of a component which is miscible in its molten state with the organic polyol ester and is more polar than the organic polyol ester component (A), at least one of the organic polyol ester and the more polar component being miscible with the organic liquid.
6. A foam control composition according to any of Claims 1 to 5, characterized in that the additive composition is present at 20-200% by weight based on the organic liquid.
7. A foam control composition according to any of Claims 1 to 6, characterised in that the branched siloxane resin consists of monovalent

trihydrocarbonsiloxy (M) groups of the formula $R''_3SiO_{1/2}$ and tetrafunctional (Q) groups $SiO_{4/2}$ wherein R'' denotes an alkyl group and the number ratio of M groups to Q groups is in the range 0.4:1 to 1.1:1.

8. A foam control composition according to any of Claims 1 to 7, characterised in that the branched siloxane resin is present at 2 to 10% by weight based on the organic liquid.
9. A foam control composition according to any of Claims 1 to 8, characterised in that the particulate filler is a silica filler with an average particle size of from 0.5 to 30 μ m.
10. A foam control composition according to any of Claims 1 to 9, characterised in that the particulate filler is present at 2 to 10% by weight based on the organic liquid.
11. A granulated foam control agent comprising a foam control composition according to any of Claims 1 to 10 supported on a particulate carrier.
12. A granulated foam control agent according to Claim 11, characterized in that a water-soluble or water-dispersible binder is also deposited on the carrier particles.

